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The dependence of the elastic properties of silica/alumina materials on the conditions used for firing

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Abstract

The dependence of the elastic properties of a range of powder compact samples has been measured as a function of firing variables. It was found that both Young's modulus and Poisson's ratio are particularly sensitive to the peak temperature and the time for which the peak temperature is maintained, over a range of these variables for which density is not significantly affected. The material investigated is used industrially for the manufacture of wall tiles. Firing trials conducted in an industrially operated tunnel kiln have indicated that sufficient variation in firing conditions exists, in the cross-section of the tunnel kiln, to cause significant variation in the values of Young's modulus and Poisson's ratio of bodies fired in different positions in the kiln. Microstructural examination of bodies produced to have very similar densities but vastly different values of Young's modulus and Poisson's ratio has indicated that the dependence of Young's modulus and Poisson's ratio on firing conditions can be explained by the extent of sintering within the ceramic matrix.

Keywords: Clays; Grain growth; Mechanical properties; Sintering; Strength; Wall tiles

1. Introduction

Many techniques are available for the evaluation and characterization of ceramic materials.[1–3] The measurement of elastic properties has received much attention, and techniques available include static loading tests and dynamic ultrasonic and vibrational resonant frequency[4,5] measurements. The well known Eq. (1) (for example see, Kolsky[6]) gives the velocity of an ultrasonic compression wave in an elastic homogenous medium of density ρ, Young's modulus E and Poisson's ratio ν:

$$V_C = \left(\frac{E(1-\nu)}{\rho(1+\nu)(1-2\nu)}\right)^{\frac{1}{2}} \tag{1}$$

With reference to Eq. (1), it can be seen that to deduce one material property from the ultrasonic compression wave velocity, two others must be known. The following Eq. (2) (see Kolsky[6]) expresses the velocity of an ultrasonic shear wave in an elastic homogenous medium:

$$V_S = \left(\frac{E}{2\rho(1+\nu)}\right)^{\frac{1}{2}} \tag{2}$$

The symbols are as previously defined. It can be seen from Eqs. (1) and (2) that if both the compression wave and shear wave velocities can be measured, then Young's modulus and Poisson's ratio can be deduced. However, in many quality control applications, such as the inference of the density of a fired ceramic body from ultrasonic compression wave propagation velocity, it is often suggested that it is valid to assume that Poisson's ratio can be treated as being approximately constant, and that Young's modulus follows a cubic relationship with density.[7,8] In this idealised limit the inference of density from compression wave propagation velocity is uncomplicated. However, under more realistic conditions, the inference of material properties from ultrasonic propagation velocity measurements can be a non-trivial matter. Analysis performed by the authors, of compression wave propagation velocity versus density results for unfired and fired powder compact samples

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formed from a silica/alumina spray dried granulate system, has indicated that the elastic properties of this particular system are significantly more sensitive to the conditions used during the firing operation than is the density.

This paper describes an investigation into the dependence of Young's modulus and Poisson's ratio for this material on the firing conditions. The conditions investigated are the ramp rate, the peak temperature and the time for which the peak temperature is maintained (the 'soak'). Sets of controlled trials were performed with a laboratory furnace, followed by an industrial trial in a commercially operated biscuit tunnel kiln. The purpose of the industrial trial was to establish whether sufficient variability in the firing conditions exists in the cross-section of the tunnel kiln to significantly affect the elastic properties of bodies fired in different positions on a kiln car.

As far as the current authors are aware, studies of the effects of variability in processing conditions on final mechanical properties have not been widely reported in the literature. Sakaguchi et al.[9] reported an investigation into the dependence of Poisson's ratio and Young's modulus on firing temperature for a range of materials representative of commercially available engineering ceramics. The results of dynamic measurements made during firing revealed that, in general, Young's modulus decreased with firing temperature, whilst Poisson's ratio showed no significant trend, although it was stated that the measurement of Poisson's ratio was subject to large error. Bogahawatta and Poole[10] described an investigation into kaolinitic clay bodies with varying contents of lime addition, produced by wet moulding. Samples were fired to peak temperatures in the range of 700 to 1150 °C, with soak times of 2 h. Using a static loading technique, it was found that the modulus of rupture decreased with peak temperature for some of the compositions and increased for others. In a similar study, Kobayashi et al.[11] presented results on the effect of firing temperature on the bending strength of various porcelains. Rectangular samples produced by slip casting were fired to peak temperatures in the range of 1150 to 1400 °C in steps of 25 °C, with soak times of 1 h. The elastic modulus of the samples was found to increase with temperature up to 1275 °C, after which a decrease was observed with increasing temperature. The elastic modulus was found to follow density in a linear fashion. Knowles[12] discussed the development of hydroxyapatite with enhanced mechanical properties, and in particular the effect of high glass additions on mechanical properties and phase stability. Samples of controlled compositions were produced by compaction and subsequent firing over a range of peak temperatures. It was observed that some rise of Young's modulus accompanied increased peak temperatures, but it was stated that the increases were not statistically significant.

As well as the effect of overall pore fraction, the influence of pore shape factors and size distribution on Young's modulus in ceramic materials has been subject to empirical and theoretical investigation. Phani[13] proposed a model which relates Young's modulus to porosity and two empirical fitting parameters. Using a least square best fit method, the model was fitted to experimental data taken for four different gypsum systems and the value of the fitting parameters optimised for each. Phani proposed that because the underlying material was the same in each case, the differences in the fitting parameters between the different systems must be due to inherent structural differences in the different gypsum systems.

Phani[14] later went on to use a self consistent spheroidal inclusion theory, which models pores as randomly distributed and orientated oblate spheroids, in an attempt to include the effect of pore shape in the prediction of the elastic properties of ceramic materials. Phani adopted a single effective aspect ratio in the derivation of his model, which was intended to represent the spectrum of aspect ratios present in real materials. Phani found that reasonable correlation could be obtained between experimentally determined results of elastic properties versus porosity and the predictions of his model for various ceramic materials, by adjusting the effective aspect ratio parameter alone. Assuming that the model is a reasonable physical representation, this suggests that pore aspect ratio is a significant factor in determining the elastic properties of a ceramic.

Boccaccini and Boccaccini[15] also incorporated the effect of pore shape in the theoretical prediction of the elastic properties of ceramic materials. In an analysis similar to the one presented by Roth et al.,[7] Boccaccini and Boccaccini showed how their model for the dependency of elastic properties on pore fraction and shape could be used to predict ultrasonic compression and shear wave velocity. They compared the results of their model with the results of Phani's model[14] for 10 different materials and found that they achieved better agreement with experimental results than did Phani where actual pore shape factors were known.

In an experimental investigation, Kathrina and Rawlings[16] employed two acoustic techniques, ultrasonic velocity measurement and acousto-ultrasonics, for the measurement of elastic properties in porous MgO ceramics. They found that the ultrasonic velocity measurements, although more reproducible than the acousto-ultrasonic measurements were sensitive to changes in the bulk pore content, but not to pore shape and size distribution. The acousto-ultrasonic technique, which involved the measurement of transmitted pulse amplitude, width and decay were found to be sensitive to pore shape and size, but it was concluded that more work is required in this area.

None of this previous work has included a comprehensive investigation into the effect of all possible firing variables on Young's modulus and Poisson's ratio. In summary of the previous works discussed here, in cases where the effect of peak temperature has been investigated, it is normally over a range of temperatures such that density would be significantly affected as well as the values of mechanical moduli. The measurement of Poisson's ratio has either received little attention, or the reported values have been associated with large experimental error, for example see Ref. 9. This paper presents a study of the effect of firing variables on the evolution of both Young's modulus and Poisson's ratio for a specific material over a range of firing temperatures and times which do not significantly affect the material density.

2. Methods

Disc shaped samples (nominally 42 mm diameter and 4.5 mm thick) were produced by the compaction of a spray dried granulate used for the production of wall tile bodies by a major industrial producer (H&R Johnson Ceramics International Ltd), on the twice-fired production process. The main constituents of the powder were ball clay (44%), china clay (8%), sand (23%), limestone (10%) and pitchers (15%) which is fired rejects from internal and external sources. The samples were formed from 10.0 g of powder, and the consolidation pressure was kept constant such that all of the samples had the same dry green density of approximately 1850 kg m^{-3}. Using this relatively high density facilitated the measurement of the resonant frequencies of vibration. Preliminary experiments revealed that higher energy signals were detected from higher density samples, suggesting that the internal damping of the sample is linked to density. The samples were fired using various cycles (see the following section), and after cooling Young's modulus and Poisson's ratio measurements performed using test C1259 of the American Society for Testing and Materials (ASTM, *Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramic Materials by Impulse Excitation of Vibration*). The technique is based on earlier work by Martincek[17] and Glandus.[18] Martincek developed existing plate theory, which considers the influence of shear deformation and rotational inertia, and derived equations to give the values of the frequencies of the first two modes of flexural vibration of 'thick' circular plates. The first mode of natural vibration is 'saddle' shaped, and the second diaphragm shaped. Both Martincek and Glandus describe a two step procedure for calculating Young's modulus. Firstly, Poisson's ratio is calculated from the ratio of the resonant frequencies of the first two modes of vibration and a knowledge of the plate dimensions. Young's modulus can then calculated individually for each of the resonant frequencies of vibration, using the value of Poisson's ratio previously calculated, in equations specific to each mode of vibration. The notional true value of Young's modulus is then found from the mean of the two values. Eqs. (3) and (4) enable Young's modulus to be calculated from the first and second modes, respectively:

$$E_1 = \frac{12\pi f_1 D^2 m(1-\nu^2)}{K_1^2 t^3} \tag{3}$$

and:

$$E_2 = \frac{12\pi f_2 D^2 m(1-\nu^2)}{K_2^2 t^3} \tag{4}$$

where E_1 and E_2 are the values of Young's modulus calculated for the first and second modes of vibration, of resonant frequencies f_1 and f_2, respectively. D is the diameter of the sample, m is the mass, t is the thickness and ν is Poisson's ratio. The factors K_1 and K_2 are geometric factors, the values of which are found by interpolation from tables, in a similar manner to Poisson's ratio. The value of Young's modulus given by the mean of E_1 and E_2 is the value which is quoted throughout this paper. Generally the two values were found to be in good agreement, typically differing by less than 0.6%.

The samples produced conformed to the specified tolerances given in ASTM C1259 for the parallelism of the faces, the aspect ratio of the sample, the flatness of the faces and the circularity of the disc. With the samples supported on a suitable support structure, measurement of the natural frequencies of the first and second modes of vibration was accomplished with a commercially available resonant frequency testing device, used in conjunction with an acoustic microphone. Reproducibility of resonant frequency measurements were found to be high, typically showing less than 0.5% variation between the maximum and minimum recorded readings for a given sample. The uncertainty in the Young's modulus and Poisson's ratio measurements were estimated as being approximately 2.5 and 1.5%, respectively. Density measurements were performed with a densometer arm in conjunction with digital balances to measure the weight of the samples in mercury, and to measure the weight of the samples in air. The uncertainty associated with the measurement of density was estimated as being less than 0.5%.

3. Firing cycles

Sets of samples were fired with different cycles to allow the effect of ramp rate, peak temperature and soak time on Poisson's ratio and Young's modulus to be evaluated. For the controlled firing trials, all firing was performed with a programmable laboratory furnace. Using the technique for sample manufacture described in the previous section, preliminary experiments revealed that the reproducibility of Young's modulus, Poisson's ratio and density between samples produced using the same firing cycle was high. The results for a set of 10 samples are given in Table 1, as a result of the high level of reproducibility, the number of samples used for each of the subsequent firing trials was limited to five.

Two sets of trials were used to assess the effect of ramp rate; one set used cycles without a soak period, the other set used cycles with a two hour soak period at the end of the ramp. Three ramp rates were used −50 °C per h, 100 °C per h and 150 °C per h. In all cases these rates were maintained until a peak temperature of 1090 °C was achieved, soak periods were then applied to the relevant samples. This peak temperature and a two hour soak period were used because they are similar to the conditions used industrially.

Seven peak temperatures were considered for the assessment of the effect of peak temperature on Young's modulus and Poisson's ratio, these being 1050, 1060, 1070, 1080, 1085, 1090 and 1100 °C. The cycle used consisted of an initial ramp rate of 57 °C per h to a predetermined temperature, followed by a fixed 2 h heating stage with a ramp rate of 20 °C per h to the peak temperature which was then maintained for a 2 h soak period. Different peak temperatures were achieved by changing the temperature reached after the first stage of heating. The cycle was selected this way so that it would as closely as possible mimic the industrial cycle.

The assessment of soak period was achieved by firing sets of samples, using a cycle with a peak temperature of 1090 °C and varying the soak period. Soak periods of 0.5, 1, 1.5, 2, 3, 4, 5 and 6 h were used, as well as a cycle without a soak period. In all cases the cycle used to achieve the peak temperature of 1090 °C consisted of an initial ramp rate of 57 °C per h to 1050 °C, followed by a reduced ramp rate of 20 °C per h to the peak temperature. Again, this type of cycle was used because it closely mimicked the traditional tunnel fired industrial process.

Table 1
Reproducibility of measurement results—mean values and standard deviations are based on measurements performed on 10 nominally identical samples

	Density (kg m^{-3})	Poisson's ratio	Young's modulus (GPa)
Mean value	1754.3	0.142	16.86
Standard deviation	3.5	0.002	0.13

4. Results of the controlled firing trials

Over the range of rates studied, the results of the trials used to assess the effect of ramp rate revealed that the particular ramp rate used has an insignificant effect on the evolution of Young's modulus and Poisson's ratio. No particular trend was observed between ramp rate and the elastic properties, although marginally higher values of Young's modulus were accompanied by higher values of Poisson's ratio. The maximum variation between elastic property values was observed to be of the same order as the experimental error associated with the particular measurement. The values of Young's modulus and Poisson's ratio measured for the samples fired with cycles including a soak time were approximately 25 and 12% higher than the samples fired without a soak period for Young's modulus and Poisson's ratio respectively. For all the sample sets, density was seen to be unaffected by the firing cycle; the mean value was found to be 1741 kg m^{-3} with a standard deviation of 3 kgm^{-3}, which is within the bounds estimated for experimental error.

The results of the trials used for the assessment of peak temperature are displayed in Fig. 1 for the effect on Young's modulus and in Fig. 2 for the effect on Poisson's ratio. In all cases density was not found to be significantly affected by differences in the peak temperature; a maximum variation of 0.6% in density was measured, although no trend with peak firing temperature was observed.

The results of the trials for the assessment of the soak period are displayed in Fig. 3 for the effect on Young's modulus and in Fig. 4 for the effect on Poisson's ratio. Again, density was not found to be significantly affected by differences in the soak period.

5. Industrial trial

The results of the controlled firing trials indicated that both Young's modulus and Poisson's ratio are sensitive to peak firing temperature and soak time, over a range of conditions for which density is not significantly affected. In industrially operated tunnel kilns, bodies are fired on kiln cars, which travel through the kiln. It is possible that different positions on the kiln car are exposed to different firing conditions. A trial was therefore conducted at H&R Johnson Ceramics International Ltd to assess whether local differences in firing conditions are indeed sufficient to significantly affect Young's modulus and Poisson's ratio.

Sets of unfired disc shaped samples, produced using the technique previously described, were positioned at different locations on a kiln car loaded with tiles. A standard kiln car was used, with 32 stacks of tiles, each stack containing 224 bodies. At the time of writing the

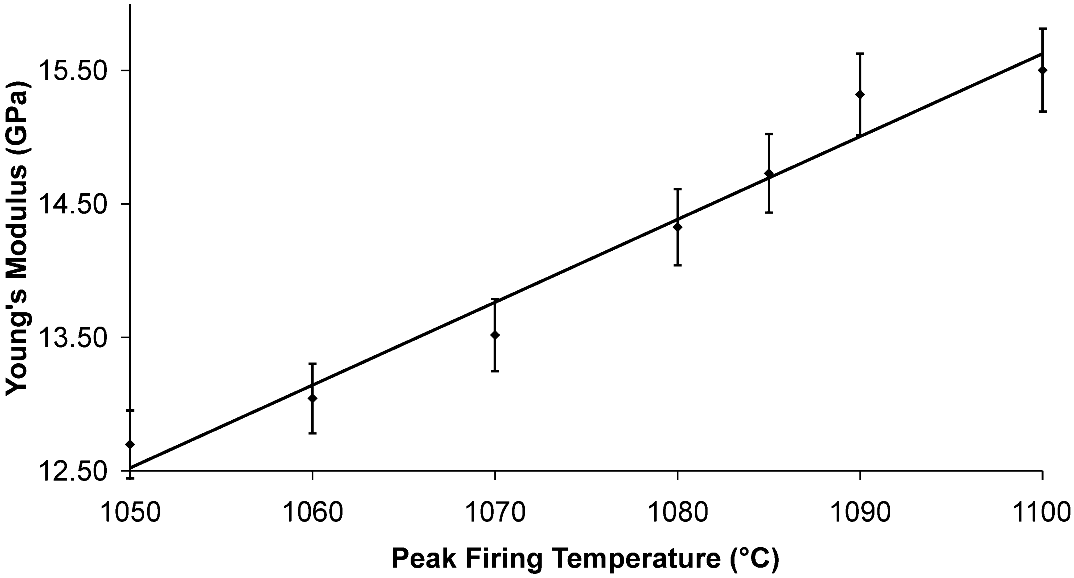

Fig. 1. Young's modulus as a function of peak firing temperature. The error bars represent ±2% of the data point values.

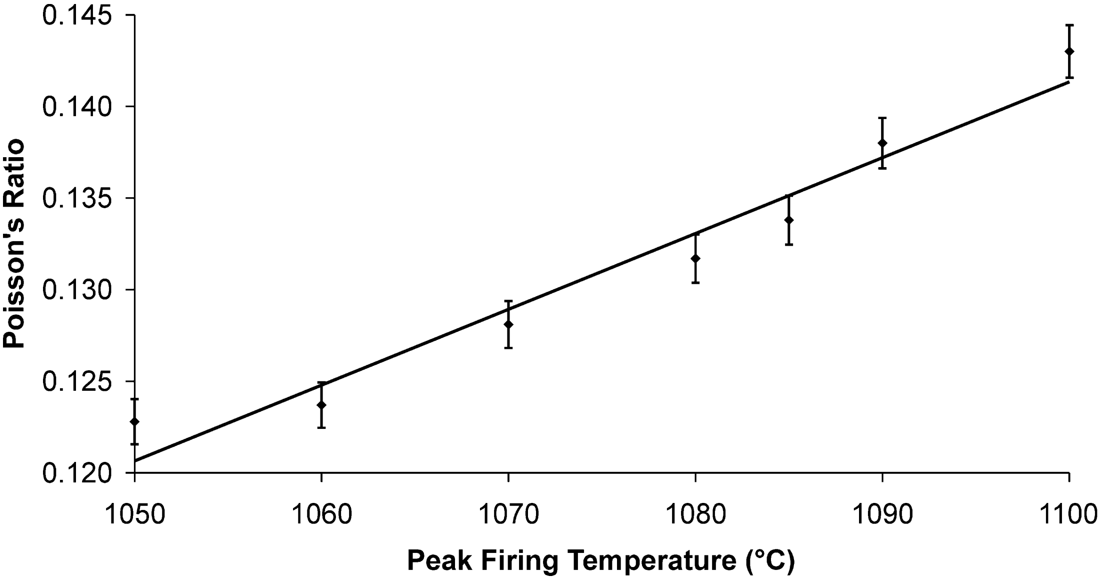

Fig. 2. Poisson's ratio as a function of peak firing temperature. The error bars represent ±1% of the data point values.

kiln cars were loaded 4 stacks wide by 8 stacks along the length of the car. One set was placed on top of the tile stacks, designated position 1. Two sets were placed approximately two thirds up the car, balanced on a support layer between the tile stacks. One of these sets was placed on the edge of the car between the outermost stacks (position 2), the other set was placed amongst the innermost tile stacks (position 3). Two more sets were placed on the base of the kiln car; again one between the outermost stacks (position 4), and one set amongst the innermost stacks (position 5). Each sample set contained three samples. After firing, the bodies were removed and elastic property and density measurements performed using the techniques described above.

No significant trend was observed between density and firing position, the mean value was found to be 1737 kg m^{-3} with a maximum variation of 0.8%. Statistically significant differences were observed for the measured values of Young's modulus and Poisson's ratio of samples fired in different positions. The specific values can be seen in Table 2.

The maximum variation in Young's modulus and Poisson's ratio seen between the firing positions are approximately 27 and 15%, respectively. The largest values of Young's modulus and Poisson's ratio were observed for samples fired on the base of the kiln car between the innermost stacks, whilst the lowest values were seen for samples fired on the outside edge of the

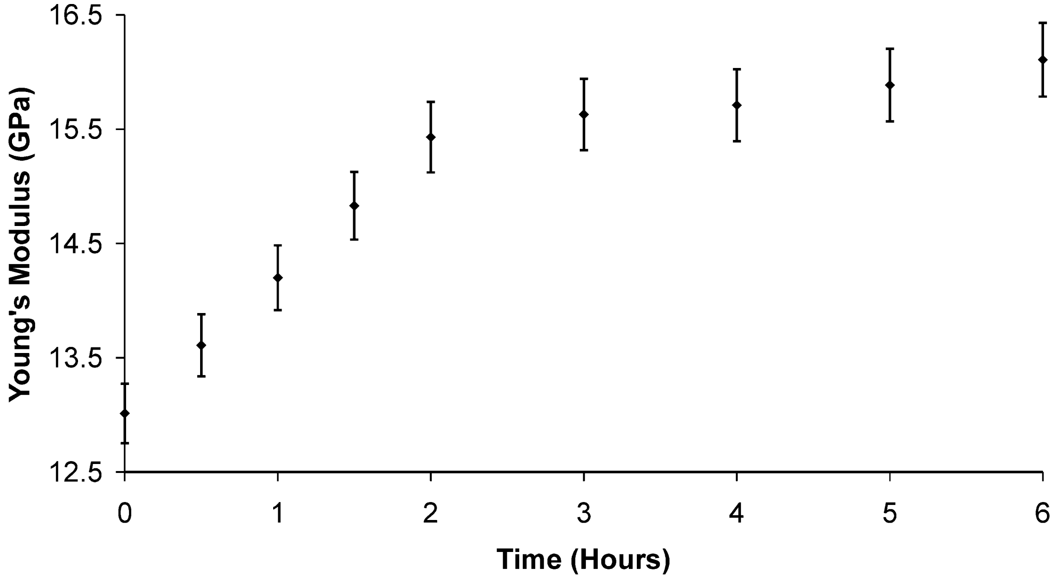

Fig. 3. Young's modulus as a function of the duration of the soak period, peak temperature 1090 °C. The error bars represent ±2% of the data point values.

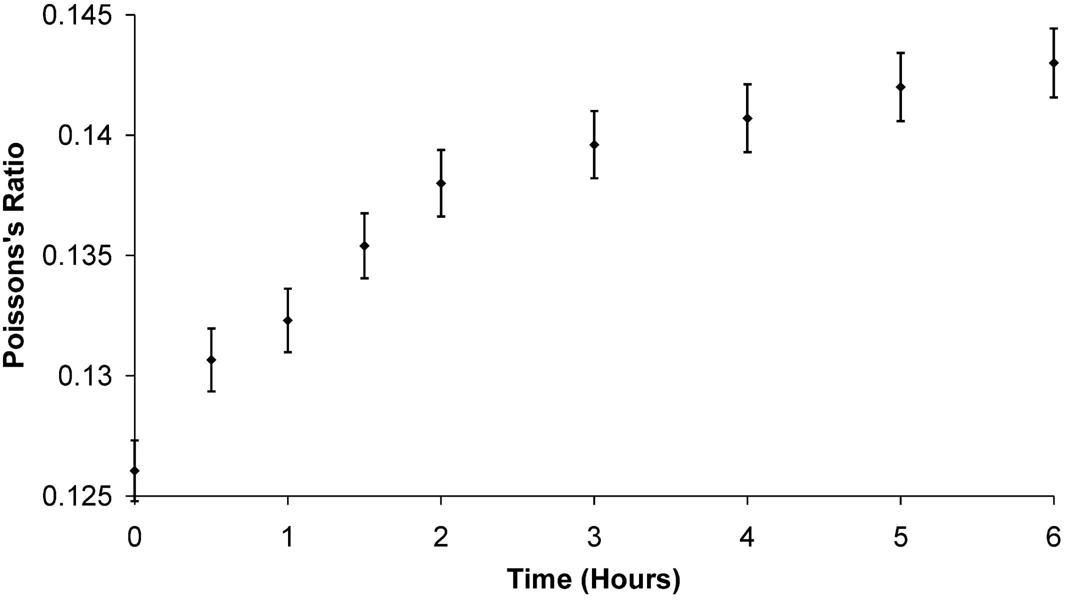

Fig. 4. Poisson's ratio as a function of the duration of the soak period, peak temperature 1090 °C. The error bars represent ±1% of the data point values.

Table 2
Values of Young's modulus and Poisson's ratio as a function of firing position in the kiln car. Values are means from 3 samples in each position

Position	Young's modulus (GPa)	Poisson's ratio
1	14.6	0.135
2	14.2	0.131
3	15.5	0.148
4	18.1	0.150
5	18.6	0.152

support layer. It is suggested that the base of the kiln car represents a large thermal reservoir. After heating, tile bodies in the vicinity of the base will cool down more slowly than tile bodies not in close proximity to the base. The tile bodies in the vicinity of the base will undergo a greater effective soak period, leading to higher values of Young's modulus and Poisson's ratio. Examination of thermographic data for a tunnel kiln of the type used in this study has shown that the peak temperatures reached at the base of the kiln car and the crown of the kiln are approximately the same. However, the temperature at the base of the kiln car after the peak temperature has been reached lags the temperature of the crown by about 2 h, resulting in an increased effective soak time. The largest values of Young's modulus and Poisson's ratio for the samples fired on the base of the kiln car were seen for the sample set which was positioned between the innermost tile stacks on the kiln car. The same is true for the samples fired on the support layer—the larger values being for the samples fired between the innermost stacks. Employing an argument similar to the one previously proposed, it is suggested that the samples positioned between the innermost stacks were in an area with a higher effective local heat capacity, and thus undergo greater effective soak periods.

6. Discussion

The sample set produced to assess the effect of a soak time of two hours underwent a similar firing cycle to that of the set of samples produced to assess the effect of a peak temperature of 1090 °C. The difference in density for these sample sets was less than the measurement error, as was the difference in Young's modulus and Poisson's ratio. This has numerous implications; the technique used for sample manufacture is accurate and reproducible, the kiln controller is reliable and the technique for the measurement of Young's modulus and Poisson's ratio are reproducible. The similarity seen between the results for the above mentioned samples and the results for the industrial trials indicates that the correct temperature range was investigated, and that the firing cycles used in the controlled trials were indeed a good approximation to the cycles used industrially. The actual variation in the firing cycle found in the cross section of the industrial kiln, be it due to peak temperature or soak time differences, can not be deduced directly. It was seen in the controlled firing trials (see Figs. 1–4) that the effect of varying the peak temperature and the soak time on Young's modulus and Poisson's ratio resulted in similar magnitude changes for the range of variation expected to be seen in the industrial tunnel kiln.

In order to establish possible reasons for the high sensitivity of Young's modulus and Poisson's ratio on firing conditions, samples were prepared for microscopic examination. Two sets of samples were produced, a set of samples with a density of approximately 1735 kg m^{-3} and Young's modulus of 16.2 GPa and a second set of four samples with the same nominal density but Young's modulus of 12.6 GPa. The different values of Young's modulus were achieved by firing the two sets of samples to different peak temperatures. The samples

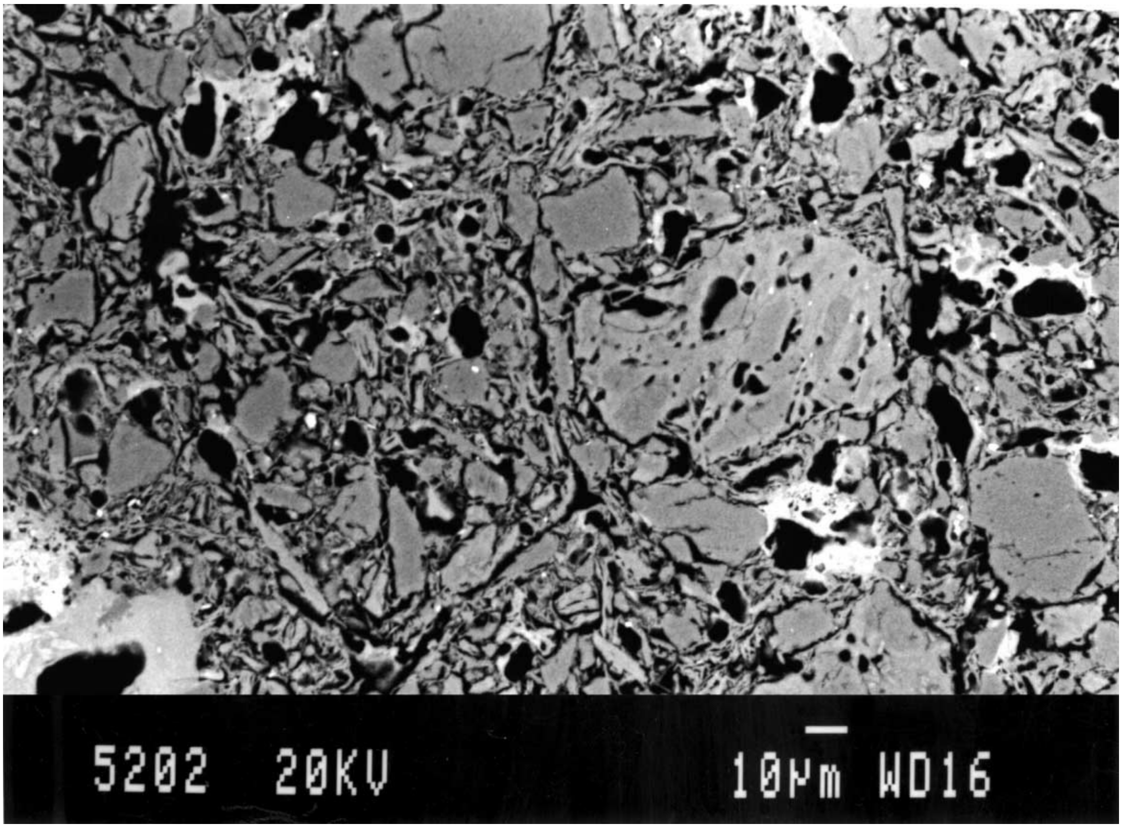

Fig. 5. Typical microstructure found in low Young's modulus samples (12.6 GPa): 500× magnification.

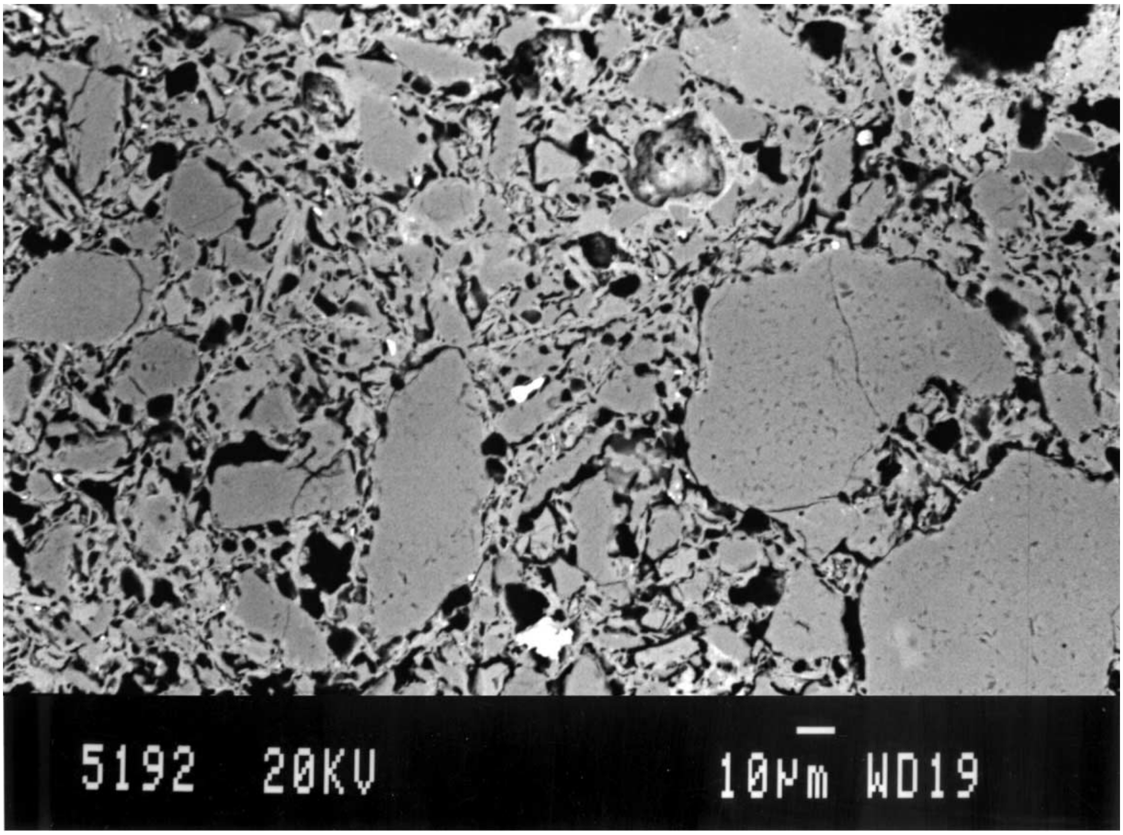

Fig. 6. Typical microstructure found in high Young's modulus samples (16.2 GPa): 500× magnification.

were sectioned and prepared for scanning electron microscopy. Micrographs were captured from areas selected at random from each sample at a magnification of 500x. Typical micrographs for the high and low modulus material are shown in Figs. 5 and 6, respectively.

From the microstructures, it is evident that a greater degree of bonding has occurred in the matrix for the higher modulus sample, suggesting that over the range of conditions investigated, the body underwent the primary stage of sintering. The curved form of the plots (Figs. 3 and 4) for the dependence of Young's modulus and Poisson's ratio on soak time indicates, for this material at a peak temperature of 1090 °C, that after approximately three hours, this stage of sintering nears completion.

7. Conclusions

Using a vibrational resonance technique, it has been shown that Young's modulus and Poisson's ratio of an industrially used silica/alumina material are particularly sensitive to the peak temperature and soak time used for the firing operation, over a range of variability found in the industrial process. Furthermore, it has been seen, that using this particular technique, reproducible results can be obtained for Poisson's ratio as well as Young's modulus. In this work it has been shown that fired bodies with very similar densities can have vastly different elastic properties.

For the particular material reported in this paper, it was observed that increases in peak temperature and soak time both resulted in higher values for Young's modulus and Poisson's ratio. Previous works[9–11] have indicated that this is not always the case. Differences here are attributed to the stage of the sintering process. In this work there is evidence to suggest that only the first stage of sintering has occurred. In these previous investigations there is evidence to suggest that later stages of sintering have occurred such as pore enlargement which can lead to a reduced strength material. Future work would therefore involve investigating the evolution of Young's modulus and Poisson's ratio over an increased temperature and soak time range such that changes brought about by more advanced stages of sintering could be investigated.

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